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MATERIALS EXPLOSIVES DIVISION

A.R.D.E. MEMORANDUM (MX) 68/60

The shatter of high strength aluminium alloys

Eleventh Progress Report

The effects of interrupted quenching on five
alloys made to specification DTD 687A

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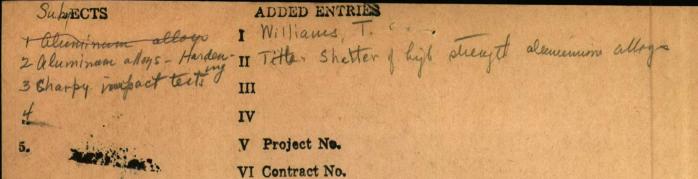
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#### The War Office

#### ARMAMENT RESEARCH AND DEVELOPMENT ESTABLISHMENT

## A.R.D.E. MEMORANDUM (MX) 68/60

The shatter of high strength aluminium alloys

Eleventh Progress Report

The effects of interrupted quenching on five
alloys made to specification DTD 687A

T. Williams (M1)

#### Summary

The relationship between the temperatures at which the solution treatment quench was interrupted for short periods and the respective hardness values obtained at definite intervals after the final cold water quench, was found to be a curve consisting of a series of promontories and bays. An alloy containing 5.4% Zn, 2.5% Mg and 0.5% Cu showed three promontories whereas four other alloys, containing about the same amount of zinc, 1.25% Cu, between 2.1 and 2.6% Mg and with two of the alloys containing 0.17% Cr and 0.05% Cd respectively, showed four such promontories, which, it was considered, indicated a four-stage ageing sequence. This was believed to be:-

GP1 zones ----> GP2 zones ----> Intermediate precipitate ----> Final precipitate

It is known that an alloy of this type containing 0.5% Cu ages in a threestage sequence, there being only one GP zone phase.

As a heat treatment for producing shatter-free alloys, there was no advantage in the use of low interrupted-quench temperatures followed by ageing at 110°C for 48 hours, but interrupting the quench at 380°C and over, and ageing similarly, resulted in improved -196°C Charpy impact values for the same hardnesses obtained by the normal treatment of DOP and FSP alloys, containing 5.5% Zn, 1.25% Cu and 2.1 and 2.5% Mg respectively. The other materials showed no improvement in properties when interrupted quenching was used, indeed it was not found possible by any form of treatment to produce freedom from shatter in the alloy containing 0.5% Cu.

Tests confirmed that small amounts of chromium had a definite weakening effect on tensile properties when interrupted quenching at 190°C, followed by ageing at 110°C for 48 hours was used, and they also showed that the presence of small amounts of cadmium caused similar behaviour.

Approved for issue:

L. Northcott, Principal Superintendent 'MX' Division

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#### 1. INTRODUCTION

A heat treatment technique has been developed by High Duty Alloys Ltd. with the primary object of improving stress corrosion resistance and minimising internal stresses in forged components of Hiduminium RR77, which is equivalent to chromium-free DTD 683 material. This heat treatment involves quenching into molten salt at 180°C from the solution treatment temperature, holding for four minutes, followed by the normal water quench and artificial ageing treatment. It was stated that an interrupted quench into molten salt reduced the tensile properties of Cr-bearing alloys to a considerable extent even when the chromium content was as low as 0.05%.

It was not known whether this treatment would produce shatter-free alloys or would have any effect on the ageing treatment recommended for the freedom from shatter of moderately alloyed material of this type i.e. 48 hours at 110°C (1). Shatter may be defined as "The catastrophic cracking of target sheets, held in tension by a stress of 30% of the tensile strength, when subjected to the blast of a 20mm H.E. cannon shell exploded on contact with a thin plate 13 inches in front of the sheet". It has been shown that for shatter-free alloys the average value of 2 longitudinal and 2 transverse Charpy impact tests of 10 s.w.g. thick material at -196°C must not be less than 0.6 ft 1b (2).

Two small sheets of clad RR77 material which had received the interrupted quench treatment were supplied by High Duty Alloys Ltd. for low temperature impact testing. The results, given in Appendix I show that the alloys were shatter-prone, and but little improvement took place on re-solution treatment at 465°C and ageing at 110°C for 48 hours. Accordingly the recommended interrupted-quench treatment was applied to the alloys DOP and FSP, containing 5.6% Zn, 2.1% Mg, 1.7% Cu and 5.4% Zn, 2.5% Mg, 1.7% Cu respectively, and although the normally shatter-free DOP material had a -196°C Charpy impact value greater than 0.6 ft lb., the FSP alloy had very poor impact properties. Other tests were carried out at various intermediate quenching temperatures without success, and it became obvious that systematic and fundamental work on the effect of this treatment would be necessary before further attempts were made to obtain shatter-free alloys by means of this type of heat treatment.

#### 2. MATERIALS

Initially it had been decided to use the alloys DOP and FSP for this investigation, but as sheet materials containing 0.17% chromium and 0.55% copper became available it appeared desirable to use these also, and to include an alloy containing 0.05% cadmium. The chemical compositions of these five materials are given in Table I.

#### 3. INTERRUPTED QUENCHING EXPERIMENTS

In view of the lack of knowledge concerning the behaviour of this type of material when the solution treatment quench was interrupted, a series of tests was planned to apply this treatment over the range 465°C to 20°C, and to allow periods of 2, 4 and 8 minutes at the intermediate temperatures.

The alloys were solution treated at 465°C for one hour, quenched into molten salt or oil at the required temperature, held for the desired period and then cold water quenched. One face of each test piece was prepared for hardness testing before heat treatment, so that only a light polish on fine emery paper moistened with paraffin was necessary, after the treatment, to enable hardness tests to be carried out. Hardness (H.V.5) determinations were made 15 minutes after the final water quench, and at intervals for a period of one week. The test pieces used were pieces of 10 s.w.g. sheet 1½ inches long and

<sup>3</sup>/<sub>4</sub> inch wide with a small hole drilled at one end for suspension, by thin wire, into the bath at the intermediate temperature. All tests were carried out in duplicate, and on separate occasions.

On plotting the interrupted quench temperature against the hardness, the resulting curve consisted of a series of promontories and bays. Figures 1 and 2 show the curves of DOP and FSP alloys obtained after 2, 4 and 8 minutes at the intermediate temperatures, the hardness values being measured 15 minutes after the water quenching operation, while figures 3 and 4 show the curves for the same materials after 1 and 2 days natural ageing. It is evident that natural ageing for 2 days obscured the changes occurring below 200 C. The curves for the chromium, low copper and cadmium bearing alloys after 4 minutes at the temperature of the interrupted quench, hardness tested 15 minutes after water quenching, are shown in Figure 5. They closely resemble those of the DOP and FSP materials similarly treated, except for the alloy containing 0.5% copper which did not show the promontory and corresponding bay occurring in the other materials between about 120 and 170 C. This alloy also showed an upper promontory at 320 C, which was rather higher than that at which the equivalent promontories of the other four materials occurred.

#### 4. INCUBATION VALUE DETERMINATIONS

The graphs representing the relationship between the temperature at which the quench was interrupted and the hardness value associated with it, appeared to have the form of a series of intersecting curves. Similar intersecting curves have been obtained by Polmear (3) by plotting incubation values against the reciprocal of the absolute temperature. The terminology introduced by Hardy (4) has become conventional for describing ageing curves. When the onset of age-hardening is delayed, the time interval involved is known as the "incubation" period. The "incubation" value is the time interval obtained by extrapolating back the rising age-hardening curve to the hardness value obtaining during the "incubation" period. Hardy considered that each C-curve represented a distinct change in the ageing process, and that the temperature of intersection represented the condition for equal probability of formation of each phase.

4.1 Specimens of DOP and FSP materials,  $1\frac{1}{2}$  inches long,  $\frac{3}{4}$  inch wide and 10 s.w.g. thick, were used to determine the ageing curves associated with the temperatures of interest. The test pieces were solution treated at  $465 \pm 3^{\circ}$ C, quenched into water at  $18^{\circ}$ C  $\pm 2^{\circ}$ C and immediately placed in ageing baths which were controlled to better than  $\pm 2^{\circ}$ C. Hardness tests (H.V.5) were carried out after various ageing periods at the required temperatures and the incubation values determined. Tests were made to find the time taken for the specimens to reach the ageing temperature, and this period was deducted from the incubation value. At certain temperatures incubation values were not observed as the alloys started to age-harden immediately on achieving the bath temperatures.

On plotting incubation values against the reciprocal of the absolute ageing temperatures, a series of intersecting C-curves was obtained, Figure 6, the temperatures of the intersections of which agreed very closely with those of the promontories found in the interrupted quenching experiments. It would seem most likely therefore that these two sets of experiments indicate the same phenomena, i.e. the different stages in the ageing sequence.

#### 5. MECHANICAL PROPERTIES ASSOCIATED WITH INTERRUPTED QUENCHING

Tensile and Charpy impact test pieces were solution treated at 465°C for one hour, quenched into molten salt or oil at the required intermediate temperature, held for 4 minutes, water quenched and immediately aged at 110°C for 48 hours.

- 5.1 The results of tensile tests on all five alloys following interruption of the quench between 190°C and 360°C and ageing as described above, are given in Table II, and the average of longitudinal and transverse tests are plotted in Figure 7. Although the general shapes of the curves of 0.1% proof stress and maximum stress were similar for all five alloys, the tensile properties of those containing small amounts of Cr and Cd suffered greater degradation than the other alloys when the quench was interrupted below 300°C. At and above this temperature the tensile properties of the alloys were very similar. Interrupting the quench at 190°C had virtually no effect on the stress values of DOP, FSP and the low Cu alloy, and at 250°C the reduction in tensile properties of these alloys was only about one third of that of those containing Cd and Cr. The lowest tensile properties of the alloy containing 0.5% Cu were obtained on interrupting the quench at 320°C, whereas the other four alloys had their lowest properties after the quench had been interrupted at 300°C.
- Hardness tests showed the same pattern as the tensile tests. The values for DOP, FSP and the low Cu alloy remained constant, when the quench was interrupted in the range 20°C to 190°C, whereas the hardnesses of the other two alloys fell continuously and at an increasing rate as the temperature at which the quench was interrupted approached 300°C. With the exception of the alloy containing 0.5% Cu, the lowest hardnesses were reached when the quench was interrupted at 300°C. The hardness of the low Cu alloy reached a minimum at 320°C.
- Charpy impact tests were carried out at -196°C, and the average of two longitudinal and two transverse test results of each material, and their hardnesses, are shown in Figures 8 and 9, plotted against the interrupted quench temperature. The behaviour of DOP, FSP and the Cr and Cd-bearing alloys was similar, having the greatest impact value after a quench interruption at 300°C. There was, however, a change in the shape of the curves between the interrupted quenching temperatures of 360°C and 465°C. The low Cu alloy possessed its maximum impact value after interruption of the quench at 320°C. The Charpy impact values of this material were considerably lower than those of the other four alloys for all intermediate temperatures.

#### 6. DISCUSSION

- 6.1 A search of the literature has revealed only two papers dealing with the effects of interrupted quenching of light alloys. Renouard (5) has determined time-temperature-property diagrams for duralumin alloys by quenching into baths at intermediate temperatures for various periods. Fink and Willey (6) carried out tests on 755 material, which is very similar in composition to DTD 687A, to determine the critical quenching rate, by interrupting the quench at various temperatures. They found that the tensile properties were adversely affected when the quench was interrupted at temperatures between 290°C and 400°C, the most critical temperature being 350°C. These results are not in complete agreement with those obtained in this investigation, which has shown that the tensile properties were adversely affected between interrupted quench temperatures of about 190°C and 400°C, the most weakening intermediate temperature being in the region of 300°C. There does not appear to have been any work carried out on the effect of interrupted quenching of this type of alloy over the whole range of temperature from 20°C to the solution treatment temperature.
- 6.2 Incubation values could not be determined above about 250°C because the test pieces started to harden in too short a time. At 320°C alloys DOP and FSP showed a small reduction in hardness after one hour's ageing, presumably due to some re-solution of the incoherent final precipitate.

The temperatures of the promontories in the curves of interrupted quench temperature versus hardness agree closely with those given by the intersections of the C-curves derived from incubation value determinations, and almost certainly indicate different stages in the ageing sequence.

Except for the low copper alloy which showed 3 promontories, the other materials containing over 1% Cu, indicated that ageing of this type of alloy occurs in 4 stages. Thomas and Nutting (7) found a three stage ageing sequence in a 0.5% Copper alloy of DTD 687A material, the sequence being CP zones—intermediate precipitate—) final precipitate.

The effect of increasing the Cu content is to cause an additional transition phase to be formed, and the curves of interrupted-quenching temperature against hardness indicate that this new phase occurs at a lower temperature than the earliest-formed phase in the 0.5% Cu alloy. The ageing sequence for alloys containing about 1.25% Cu is therefore believed to be:-

GP1 zones GP2 zones Intermediate precipitate Final precipitate.

It is possible that the GP zones observed by Thomas and Nutting are the GP 2 zones in the sequence above.

The four-stage ageing sequence described is similar to that found to occur in the Al-Cu alloys of the duralumin-type

The effects caused by interrupting the quench from the solution treatment temperature are due, in part, to the reduction of the quench-induced stresses. As in "martempering" of ferrous alloys, interrupting the quench at some intermediate temperature for sufficiently long to allow temperature equalisation results in lower stresses arising from the final quench. Below 300°C, in addition to the stress-reducing effect, nuclei of the particular GP zone or precipitate in equilibrium at that temperature are also formed. At 300°C precipitation is complete, (320°C for the 0.5% Cu alloy), and as this is incoherent with the matrix, the materials are soft and have enhanced shock resistance. Above 300°C the final precipitate redissolves in the matrix, and re-solution is sensibly complete by about 380°C. For the low Cu material these temperatures are about 20°C higher.

The reduction in stress level is perhaps illustrated by the greater -196°C Charpy impact values with satisfactory hardnesses of DOP and FSP alloys obtained when the quench is interrupted at 380°C or over. Although the Cr and Cd-bearing materials would have satisfactory shatter resistance, both the -196°C impact values and hardnesses are slightly lower than those obtained by conventional treatment. The alloy containing 0.5% Cu could not be made to combine freedom from shatter with the required tensile strength, by any form of treatment.

- 6.5 The tensile properties of the DOP and FSP materials, and of the low Cu alloy suffered only slight degradation when interrupted quench temperatures of up to about 200°C were used, whereas these properties of the alloys containing small amounts of Cr and Cd suffered marked reduction even at an intermediate temperature of 150°C. These results confirm the observations of High Duty Alloys Ltd. on the deleterious effect of Cr on the tensile properties after interrupting the quench at 180°C, and also indicate that in this respect Cd behaves similarly.
- 6.6 The table below shows the interrupted quench temperatures which may be used, followed by ageing at 110°C for 48 hours, to produce shatter-free material i.e. material having a -196°C Charpy impact value of not less than 0.6 ft lb with a hardness (HV 5) greater than 175.

Material	Temperature of quench interruption
DOP FSP with Cd with Cr with 0.5% Cu	up to 200°C and above 360°C up to 80°C and above 360°C up to 80°C and above 370°C up to 80°C and above 370°C NONE

The first four alloys in this table can be brought into a shatter-free condition by ageing at 110°C. It might therefore have been thought that interrupting the quench at 110°C, followed by ageing at the same temperature, would also have produced satisfactory material, but the results above do not support this expectation, except in the case of DOP material, containing only 2.13% Mg, which is also shatter-free after ageing at 135°C.

On quenching from 465°C into an oil bath at 80°C a moderately agitated test piece cooled through the suggested GP2 range, i.e. from 175°C to 120°C, in 2.8 secs whereas when the oil bath was at 110°C, the same specimen with the same amount of agitation cooled through this range in 8.3 seconds. It would therefore appear that about 8 secs. was long enough to have allowed formation of sufficient of the phase stable between 175°C and 120°C to interfere with the production of GP1 zones on ageing at 110°C.

Since the suggested GP1 zones are not formed in the 0.5% Cu alloy, which is shatter-prone, it would seem that freedom from shatter of moderately alloyed material can only be achieved in the presence of the GP1 zone structure.

It was shown in A.R.D.E. memorandum (MX) 66/59 that if the fracture path of Charpy impact specimens broken at -196°C was predominantly intergranular, the material was shatter-prone. Examination of the fracture path of Charpy test pieces of the 0.5% copper alloy, after re-solution treatment and ageing at 110°C for 48 hours, and broken at -196°C, showed the fracture path to be 67% intergranular.

#### 7. CONCLUSIONS

By use of an interrupted quench technique it has been found possible to determine the ageing sequence of DTD 687A alloys containing high and low magnesium and copper contents, and also small amounts of cadmium and chromium. With the exception of an alloy containing only 0.5% Cu, the ageing sequence is believed to be:-

GP 1 zones > GP 2 zones -> intermediate precipitate -> final precipitate.

In the low Cu alloy this suggested GP 1 zone phase is absent.

The temperature domains of the different phases determined in this work apply to the conditions obtaining during these experiments.

- 2. Moderately alloyed materials containing approximately 1.25% Cu can, it has been shown, be made shatter-free by ageing at 110°C i.e. by ageing in what is presumably a GP 1 zone region. It has not been found possible to devise heat treatment schemes to produce freedom from shatter in material containing 0.5% Cu.
- No advantage would be derived from the use of low interrupted—quench temperatures followed by ageing at 110°C for 48 hours in the production of shatter-free alloys. For the alloys DOP and FSP interrupted quenches at temperatures of 380°C and over resulted in improved -196°C impact values for the same (maximum) hardnesses, but the materials containing small amounts of Cd and Cr showed no improvement.
- Tensile test results after interrupted quenching and subsequent ageing at 110°C showed no degradation of properties for the materials DOP and FSP up to an interrupted-quench temperature of 190°C, whereas the Cr and Cd-bearing alloys gave low values. These results confirm the findings of High Duty Alloys Ltd. on the deleterious effect of small amounts of Cr on the tensile properties when interrupted quenching is used, and indicate that the effect of Cd is similar.

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TABLE I

Compositions of Alloys used

No. 1	-			Per	Cent			
Material	Zn	Mg	Cu	Mn	Fe	Si	Cr	Cd
DOP	5•57	2.13	1.27	0.39	0.24	0.10	<0.01	NIL
FSP	5.41	2.52	1.28	0.41	0.02	0.01	0.01	NIL
Cr-bearing	5.52	2.30	1.22	0.30	0.28	0.14	0.17	NIL
Cd-bearing	5.57	2.59	1.28	0.40	0.22	0.10	0.01	0.05
Low-Cu	5.40	2.55	0.55	0.39	0.27	0.12	<0.01	NIL

TABLE II

Tensile properties after Interrupted Quenching and ageing at 110°C for 48 hours

									-	
	豆鼠	14	172	13/2	16	15	14=	13/2	15	13
360°C	360°C U.T.S. per	33.3	33.6	33.55	32.9	32.7	33.0	32.6	34.04	33.0
	0.1% U.T. P.S. tons per sq. inch	26.9	27.3	27.0	26.2	25.8	26.2	26.2	28.2	28.0
	E1.	t	1 1	ı	1	t	1	1	15	132
320°0	.S. U.T.S. tons per sq. inch	ı	1 1	t	1	1 -	1	ı	20.6	20.4
	P.S. tons	1	t - d	1	1	ı.	t	1	10.3	10.3
	E1.	20	174	162	17	14-2	16	15	15	14
300°C	U.T.S.	22.2	22.7	22.2	21.8	21.5	22.3	21.9	23.3	22.8
	0.1% U.T P.S. tons per sq. inch	10.1	10.3	10.1	7.6	9.5	10.6	10.4	13.4	13.2
	E1.	15	13	11	142	13	132	13	15	135
250°C	U.T.S.	32.9	33.9	32.7	27.3	27.1	27.6	27.0	31.3	30.4
	0.1% U.T P.S. tons per sq. inch	25.4	25.8	25.6	16.2	16.2	15.7	15.7	24.1	24.1
	E1.	142	C 7 7	122	124	12	12	11	142	13
190°G	190°C U.T.S. per	36.3	57.0	36.2	32.5	34.8	33.9	33.4	35.2	7.42
	P.S. U.T. tons per sq. inch		29.8	29.6	24.8	24.0	25.4	25.2	28.6	28.6
it.	EI %	15	152	13	152	15	125	11	152	142
20°C(465°C)*	U.T.S. per nch	36.5	37.3	36.2	36.3	36.2	9.04	39.4	36.0	35.3
20°0	0.1% U.T. P.S. tons per sq. inch	29.7	30.3	30.1	30.0	29.8	34.3	30.7	29.6	28.8
Intermediate		DOP L	T. TST. I.	H	Cr-bearing L	EH	Cd-bearing L	EH	Low-Cu L	E

\* The tensile values for an interrupted quench temperature of 20°C are the same as those for 465°C, since quenching from the latter temperature into water at 20°C may be considered to represent interrupted quenching at either temperature.

#### APPENDIX I

# Tests of clad Hiduminium RR77 material supplied by High Duty Alloys Ltd.

Two pieces of clad Hiduminium RR77 material, approximately 12" x 6" and 10 s.w.g. thick, were received for examination.

The treatment given was said to have been: -

45 minutes at 460°C,

Quenched into salt at 180°C for 4 mins,

Cold water quenched and stretched 12% in the direction of rolling, Aged for 8 hours at 135°C.

The 12% stretch was applied because this was considered to be the equivalent of the cold flattening given to production sheets after solution treatment.

## Tensile Tests on supplied material

		Longit.	Transv.	HV (5)
0.1% Proof Stress)	tons	28.0	27.8	178
Maximum Stress	per sq.inch	31.7	31.7	
Elongation	%	12	11	, ,

# Charpy Impact tests at -196°C

Sheet (a) Longit. 0.51 0.48 ft. lb. Transv. 0.43 0.46 ft.lb.Average 0.47 ft. lb.

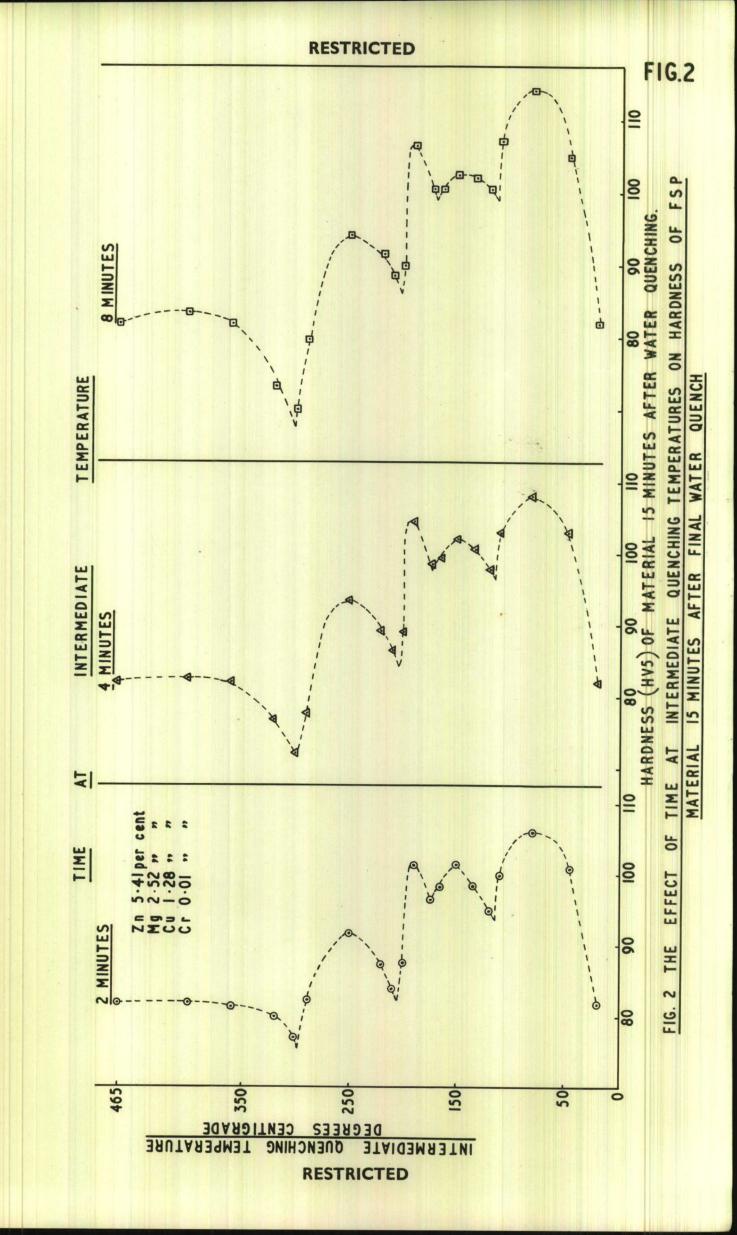
Sheet (b) Longit. 0.45 0.48 ft. lb. Transv. 0.41 0.41 ft.lb.Average 0.44 ft. lb.

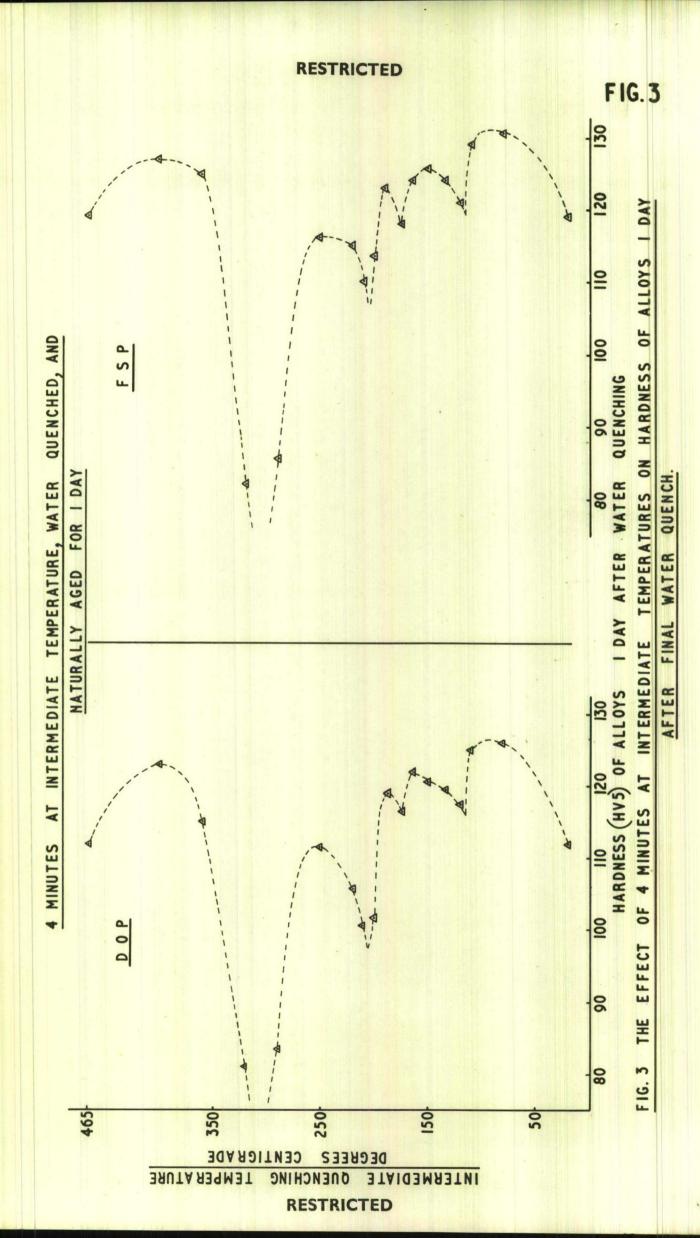
Since the material was judged from these figures to be shatter-prone, it was re-heat treated, i.e. 465°C for 6 hours, water quenched, aged at 110°C for 48 hours.

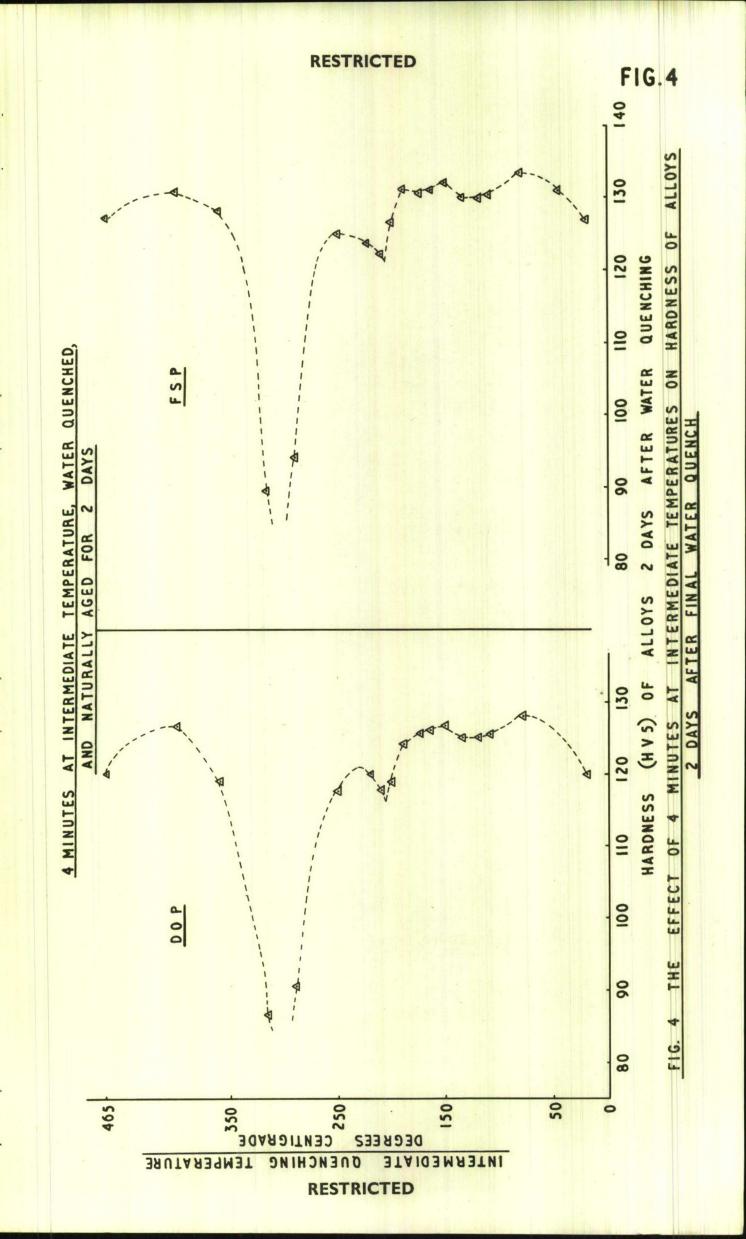
# Re-heat treated Charpy impact tests at -196°C

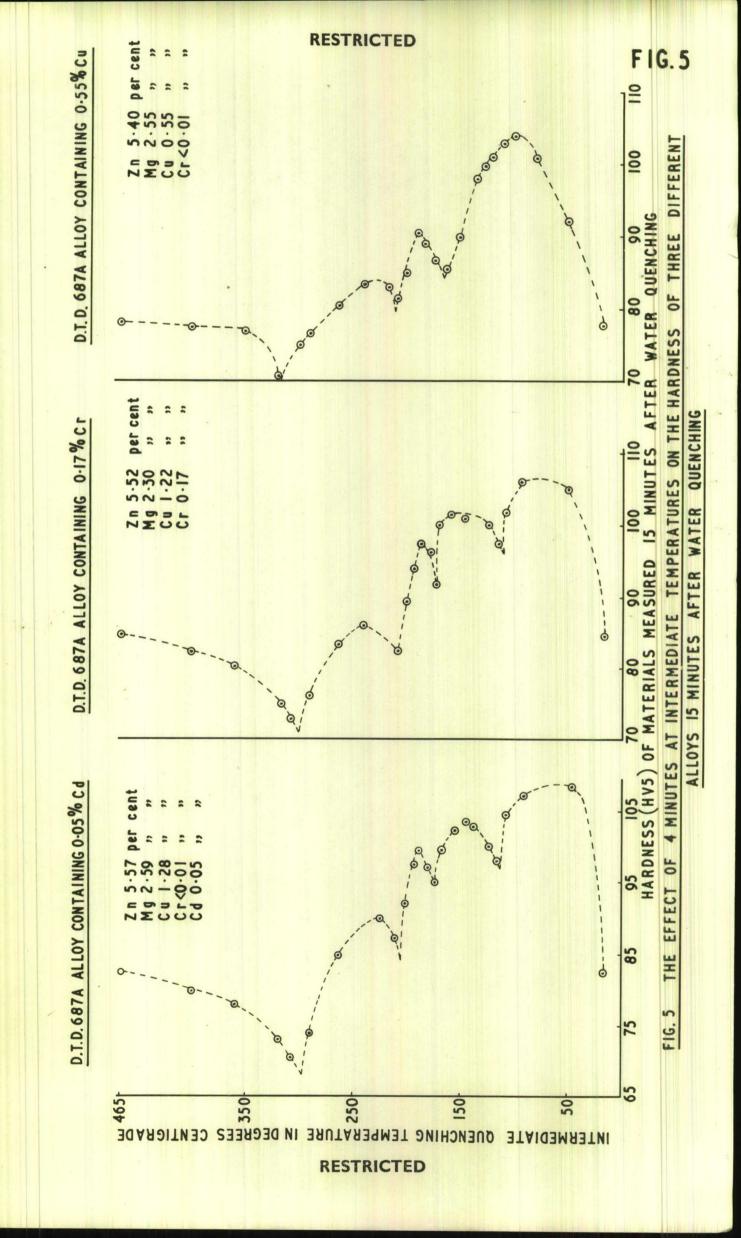
Longit. 0.52 0.49 ft.lb. Transv. 0.43 0.46 ft.lb. Average 0.48 ft.lb. HV(5) 185.

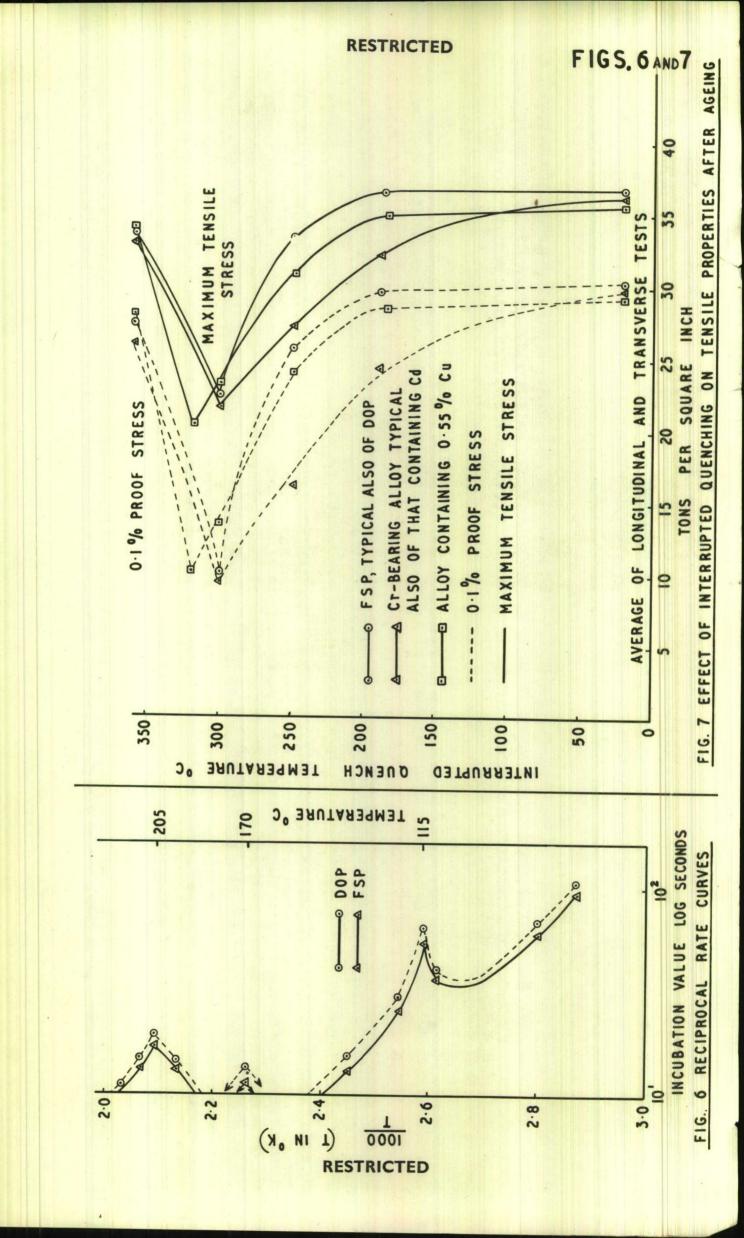
It should be remembered that RR77 material contains only 0.5% copper, and it has not been found possible to obtain such material in a shatter-free condition, with adequate tensile strength.

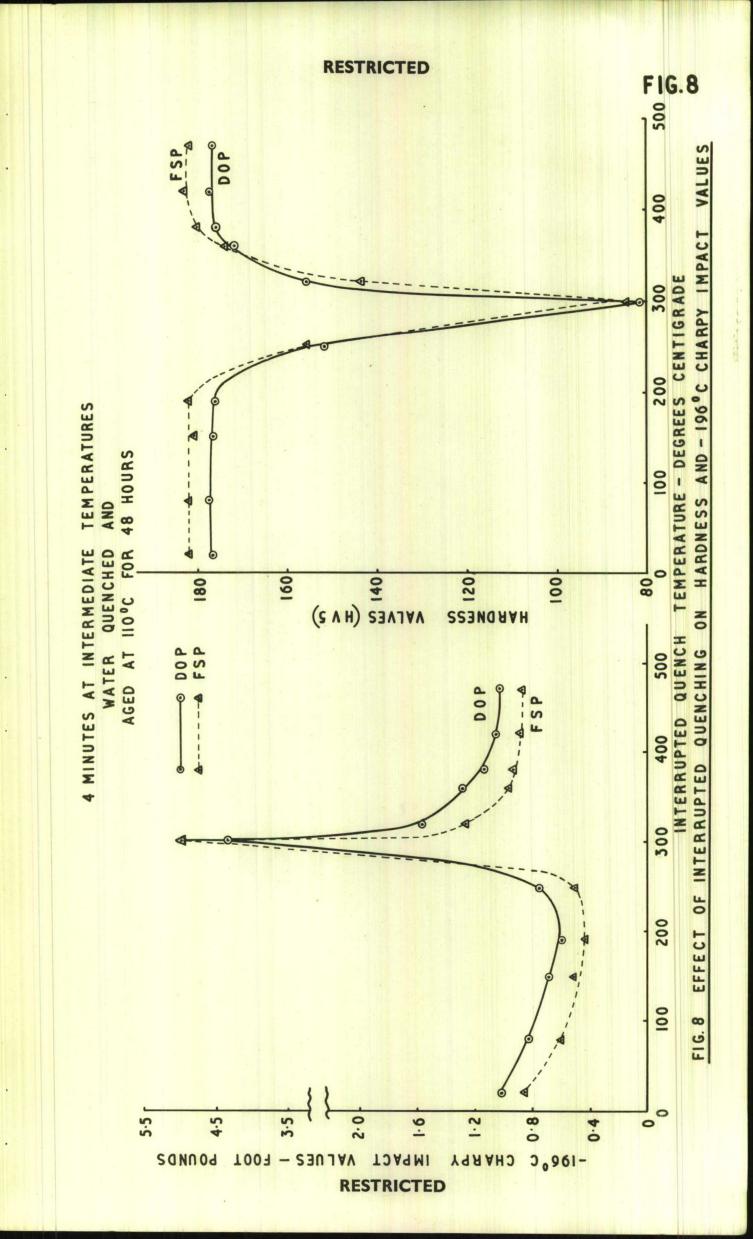


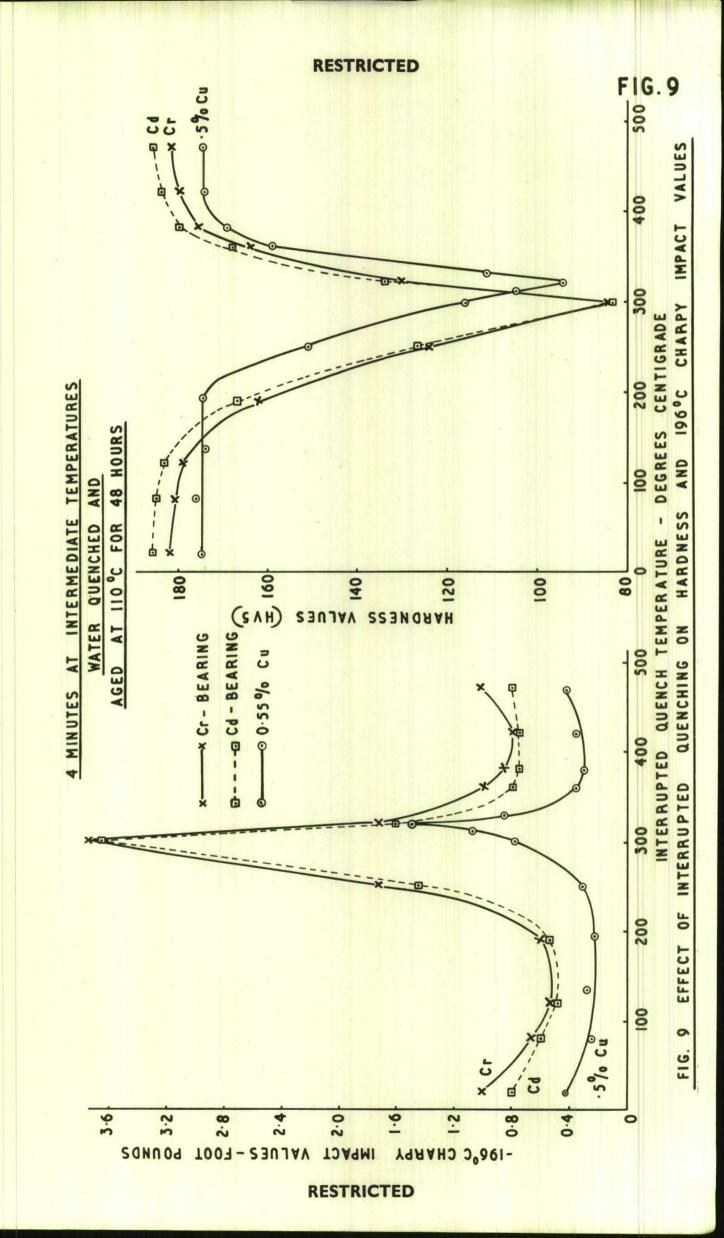












Armament Research & Development Establishment A.R.D.E. Memorandum (MX)68/60

669.715: 669.715-156: 620.178.72

The shatter of high strength aluminium alloys. Eleventh Progress Report.

The effects of interrupted quenching on five alloys made to specification DTD 687A

T.Williams.

December 1960

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GP1 zones - GP2 zones - Intermediate precipitate - Final precipitate

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It is known that an allow of this type containing 0.5% Cu ages in a three-stage sequence, there being only one GP zone phase.

As a heat treatment for producing shatter-free alloys, there was no advantage in the use of low interrupted-quench temperatures followed by ageing at 110°C for 48 hours, but interrupting the quench at 380°C and over, and ageing similarly, resulted in improved -196°C Charpy impact values for the same hardnesses obtained by the normal treatment of DOP and FSP alloys, containing 5.5% Zn, 1.25% Cu and 2.1 and 2.5% Mg respectively. The other materials showed no improvement in properties when interrupted quenching was used, indeed it was not found possible by any form of treatment to produce freedom from shatter in the alloy containing 0.5% Cu.

8pp. 9 figs. 2 tabs. 7 refs.

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